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Enhancement of Sulphur Removal Efficiency from Propane-Propylene Fraction (PPF) from Catalytic Cracking Process (CCP) by Adsorption: A Field Study

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Abstract

The removal of sulphur from propane-propylene fraction (PPF) is crucial for enhancing hydrocarbon product quality and meeting environmental standards. This study aimed to investigate the effectiveness of adsorption methods used to remove sulphur-containing compounds from PPF using different adsorbents and recovery methods. The experiment was conducted at the olefin plant of the Ethylene-Polyethylene complex in Sumgait, Azerbaijan, employing a two-stage purification process. Four types of adsorbents (A, B, C, and D) were tested. The first adsorber containing adsorbents A, B, and C removed water, mercaptans and carbonyl sulphide (COS), while the second adsorber with adsorbent D removed residual impurities of arsine, COS and hydrogen sulphide. The recovery of adsorbents A, B and C was carried out using methane at 230-250°C. The results showed that the initial sulphur concentration in the starting material, which was 5.67 ppm, decreased significantly after treatment. The two-stage purification process effectively removed both major and trace amounts of sulphur-containing compounds, which confirms the high efficiency of the selected adsorbents. The regeneration process showed its economic and technological stability, providing the possibility of multiple use of adsorbents A, B and C. Further optimization of the process parameters, including temperature and pressure in the adsorbers, will help to increase overall efficiency and reduce operating costs. The study confirmed the promising potential of adsorption methods for industrial sulphur removal from PPF, which contributes to improved product quality and environmental safety of the process.

Keywords

Dsulphurization; Zeolites; Deep purification technologies; Catalysts; Metal-oxide sorbents; Selectivity

Introduction

Liquid catalytic cracking is one of the key refining processes that helps to convert crude oil into sought-after petroleum products (Bao *et al.,* 2023). Among the obtained materials, an important one is propane-propylene fraction (PPF), which is the main feedstock for the production of polypropylene, as mentioned by Stratiev *et al.* (2023) and Gholami *et al.* (2021). However, the presence of sulphur in PPF poses a significant problem as it adversely affects polymerization catalysts, thereby diminishing the quality of the final product. Hariz, Al-Ayni and Monser (2014) state that the removal of sulphur from PPF and other hydrocarbon streams by adsorption has become one of the promising areas to improve product quality and environmental compliance.

There are various methods of desulphurization, including hydrodesulphurisation, absorption and adsorption. Saha, Vedachalam, and Dalai (2020) single out adsorption as a cost-effective and relatively easy-to-apply method that does not require significant process changes. It is important to distinguish adsorption from absorption, the latter referring to the process in which a substance is taken up into the internal volume or bulk phase of another material, whereas adsorption involves the accumulation of molecules on the surface of a solid. Among adsorption methods, zeolites and aluminium oxide have proved to be effective materials for the removal of sulphur-containing compounds from PPFs. The adsorption efficiency depends on several factors, including the type of adsorbent used, its porosity, active surface area, and process conditions such as temperature, pressure, and contact time. The most commonly used adsorbents are zeolite, activated carbon, and metal oxide-based adsorbents (Asgerov *et al.,* 2022; Lyubchik *et al.,* 2008).

Zeolites such as X, Y, and photosite exhibit pronounced adsorption properties, particularly when modified via ion exchange with transition metals. As shown by Jiang *et al.* (2021), the incorporation of metal ions such as Zn^{2+}, Cu^{+}, and Ag^{+} into zeolite frameworks substantially enhances their sorption capacity and selectivity toward sulphur-containing compounds. A critical factor underlying this performance is the hierarchical structure of the zeolites, which combines micropores and mesopores, thereby facilitating the diffusion and adsorption of sulphur molecules of varying molecular weights. In the case of adsorbent B, which comprises modified alumina with zeolite, this hierarchical porosity enables efficient capture of both small molecules like hydrogen sulphide (H_2S) and larger, less volatile species such as mercaptans.

The mesoporous channels reduce diffusion limitations, allowing for faster transport of bulky molecules to active adsorption sites located within microporous domains (Doroshkevich *et al.,* 2017; Petrov *et al.,* 2020). Similarly, adsorbent D, based on MnO_2, benefits from its micro-mesoporous structure and high surface reactivity, particularly enhancing its selectivity for carbonyl sulphide (COS), which is more challenging to remove due to its low polarity and kinetic stability. As demonstrated in the work of Dehghan and Anbia (2017), metal ion modification (e.g., with Ce^{4+} or Pd^{2+}) not only increases affinity for sulphur species but also reduces competitive adsorption of aromatics and nitrogen-containing compounds, thus preserving the adsorbent's selectivity under complex hydrocarbon matrices. The synergy between hierarchical pore architecture and tailored surface chemistry is therefore fundamental in achieving high-

efficiency desulphurization, especially when targeting structurally diverse sulphur contaminants.

Aluminium oxide, as indicated in the work of Alcaraz *et al.* (2023), is successfully used as a catalyst carrier in hydrotreating processes such as hydrodesulphurization. Activated aluminium oxide, such as GSorb-600, shows high stability and efficiency in the removal of sulphur-containing impurities at high temperatures. The approach allows efficient use of pre-adsorption by zeolites followed by catalytic hydrotreating based on aluminium oxide, which provides ultra-low sulphur content in the final products. In addition, metal-exchanged mesoporous Y zeolites, according to Lee and Valla (2016), are a promising method for desulphurization of transport fuels. The introduction of mesopores reduces diffusion limitations and improves the adsorption of large sulphur-containing molecules such as dibenzothiophene. Ce and Cu metals contribute to the selectivity towards sulphur-containing compounds, especially for thiophene molecules. The mesoporous structure of Y zeolites facilitates access to active centres, and metal cations enhance the capture of sulphur-containing molecules, making this method effective for fuel purification.

Moreover, metal-organic frameworks such as MIL-53 (Cr, Al, Fe), Cu-BDC and HKUST-1 have shown good adsorption properties for desulphurization of aromatic sulphur compounds, as shown in a study by Yoosuk *et al.* (2023). The highly active surface area and significant porosity of these frameworks make them suitable for deep sulphur removal from fuels and hydrocarbon streams. Adsorption desulphurization is one of the most promising methods of sulphur removal, as it can be implemented at relatively low temperatures and does not require significant energy costs (Dinzhos, Privalko and Privalko, 2005; Privalko, Dinzhos, and Privalko, 2005). A key aspect of the successful application of adsorption technologies is the possibility of adsorbent regeneration. Regeneration can be performed by both thermal and chemical methods, which allows restoring the adsorption capacity of the material and using it repeatedly.

Despite significant progress in the development of adsorbents for the removal of sulphur-containing compounds, several problems remain unresolved. These include limited selectivity of adsorbents in the presence of complex hydrocarbon matrices, reduced efficiency in the removal of high molecular weight sulphur compounds, and the need for stable and efficient regeneration of adsorbents for repeated use. These issues are particularly relevant for the purification of PPF, since PPF contains both light and heavy sulphur compounds. The present study is aimed at investigating the possibility of using adsorption methods for the removal of sulfur-containing compounds from PPFs obtained in the CKD process. The key objectives to achieve the aim are:

1. to evaluate the effectiveness of different adsorbents for the removal of such sulfur compounds;
2. to consider the possibility of adsorbent regeneration using a mixture of methane and hydrogen as a regeneration gas.

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Materials and Methods

Selection and Characteristics of Adsorbents

The study was carried out under industrial conditions at the olefin plant of Ethylene-Polyethylene (Sumgait, Azerbaijan) to evaluate the efficiency of sulphur removal from PPF using different adsorbents and their regeneration methods. Four adsorbents, A, B, C and D, were used in this study, chosen for their synergistic role in a two-stage sulphur removal procedure. Adsorbent A, consisting of clay-based alumina, was mainly used for water and hydrogen sulphide extraction due to its high dehydration efficiency. Adsorbent B, which was a modified alumina combined with zeolite, showed improved selectivity for water and mercaptans by utilizing the structural characteristics of the zeolite phase. Adsorbent C, consisting of unmodified alumina, functioned as a versatile sorbent for the removal of both water and sulphur compounds, but with reduced selectivity compared to B. Adsorbent D, consisting of manganese dioxide (MnO_2), was used in the second stage of purification to remove trace amounts of carbonyl sulphide (COS) and hydrogen sulphide. D, as a non-recoverable substance, acts by irreversible chemisorption, achieving deep purification when alternative adsorbents are less effective. Table 1 provides a comparative overview of their composition, functions and characteristics, providing a better understanding of their respective roles in the desulfurization process.

Table 1: Summary of Adsorbent Properties and Functional Roles

Adsorbent	*Composition*	*Primary Function*	*Performance Highlights*
A	Alumina (clay-based)	Removal of water and H_2S	Effective dehydration; moderate sulphur removal
B	Modified alumina with zeolite	Removal of water and mercaptans	High selectivity for mercaptans; enhanced water capture
C	Alumina	Supplementary removal of water and sulphur compounds	General support; less selective
D	MnO_2	Selective removal of COS and H_2S (non-regenerable)	High COS and H_2S affinity; irreversible binding

Functional Role of Non-Regenerable Adsorbent D

Adsorbent D, consisting of MnO_2, is classified as non-regenerable because its chemical interaction with sulphur compounds such as hydrogen sulphide (H_2S) and carbonyl sulphide (COS) leads to the formation of stable metal-sulphide bonds that are not easily reversible under standard regeneration conditions. Unlike alumina or zeolite-based adsorbents (A, B and C), which utilize mainly physical adsorption and can be efficiently regenerated by methane at high temperatures, MnO_2 undergoes chemisorption processes that irreversibly alter its structure and adsorption capacity. Thus, the non-recoverability of this material entails increased long-term maintenance costs associated with the need for its frequent replacement. Its inclusion in the second stage of the purification process is justified due to its remarkable selectivity and strong attraction for residual sulphur compounds, especially COS, which are more difficult to remove. Although adsorbent D

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increases material costs, its important function in achieving ultra-low sulphur concentrations guarantees compliance with industrial standards and prevents catalyst poisoning in subsequent polymerization operations.

Process Design and Equipment Setup

To ensure the continuity of the process, a parallel mode of operation of adsorbers was envisaged: one adsorber was in operation and the other was in the regeneration stage or in standby mode. Inert porcelain balls were used to ensure uniform gas flow. The balls were loaded with the adsorbents to ensure uniform distribution of gas flow through the adsorbent layers and to prevent dynamic effects associated with gas flow. Figures 1 and 2 show the process flow diagrams and the design of the adsorbers used in this study.

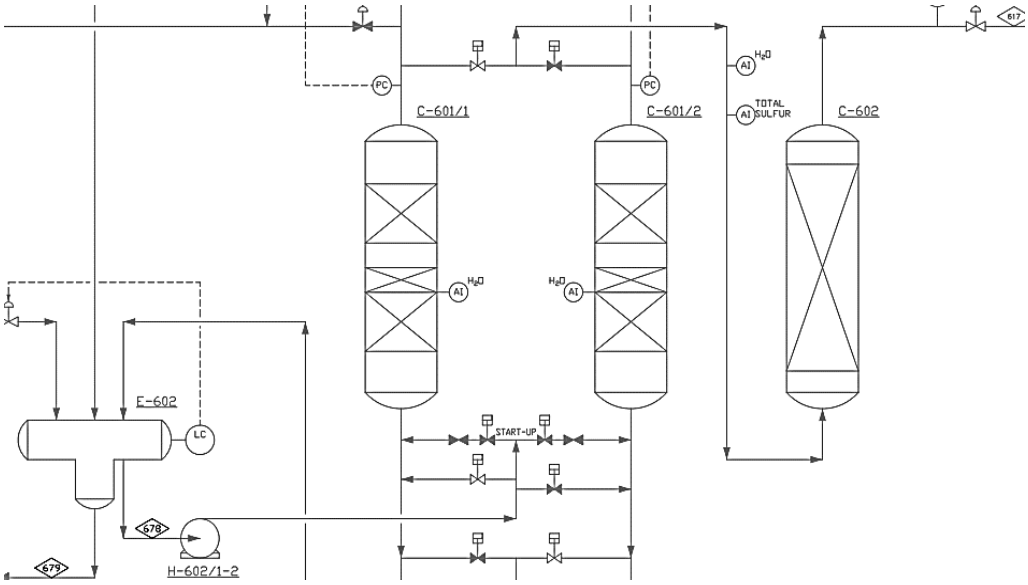

Figure 1: Simplified process flow diagram illustrating a PPF treatment plant for the removal of sulphur compounds

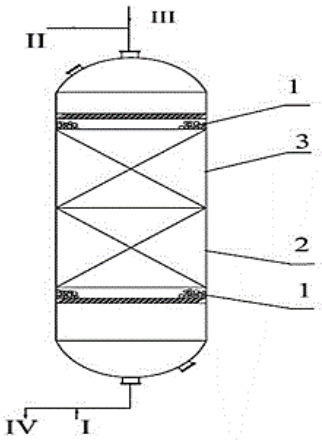

Figure 2: Schematic diagram of the adsorber used in pilot tests. Showing 1 – ceramic beads; 2 – adsorbents; I – PPF before treatment; II – PPF after treatment; III – gas regeneration and cooling; IV – gas after regeneration and cooling.

Operating Conditions and Adsorption Cycle

The feed rate of PPF through the adsorbers was set at 18 tonnes/hour. The adsorption cycle duration was 120 hours, after which the adsorbent regeneration stage began. Importantly, the use of a two-stage process allowed for more efficient removal of sulphur-containing compounds from the stream and deeper purification at each stage. Adsorbents A, B and C used in the first adsorber were selected for their regeneration capability. Adsorbent D, used in the second stage, was disposable and was used for the final purification of the PPF stream from residual impurities. This method maximized the purification of the hydrocarbon stream while minimizing the cost of adsorbent replacement.

Regeneration Procedure for Adsorbents A, B, and C

Methane was used for adsorbent regeneration due to its chemical inertness, cost-effectiveness, and compatibility with alumina-based adsorbents, allowing for efficient desorption of accumulated sulphur compounds at high temperatures without causing adsorbent degradation. Unlike hydrogen-rich mixtures, which exhibit increased reactivity and require more stringent handling protocols, methane provides sustained thermal regeneration while reducing chemical interaction with the adsorbent surface. In addition, methane is readily available at petrochemical plants, facilitating its incorporation into existing infrastructure. The regeneration of adsorbents A, B and C was carried out using methane as the regeneration gas. The process involved gradually increasing the gas temperature to 250°C at a rate of less than 50°C per hour at a pressure of 0.5 MPa. Methane was passed through an absorber at 230°C, heating the adsorbent and promoting desorption of accumulated sulphur-containing compounds, hydrocarbons, and water vapour. The gas then entered a heat exchanger where it was cooled before being fed to a separator where condensed hydrocarbons and moisture were separated. The cleaned regenerated gas was returned to the pipeline for subsequent use. The temperature of the regenerated gases was maintained at 230°C for 3 hours, after which the adsorber cooling stage began.

Analytical Methods and Sulphur Measurement

Sulphur concentration measurements in this study were performed using gas chromatography with a chemiluminescent sulphur detector (SCD), which achieved an analytical accuracy of 0.05 ppm. The initial sulphur content of PPF was 5.67 ± 0.05 ppm, but after treatment, the values ranged from 1.92 to 1.98 ± 0.05 ppm over many tests, confirming a consistent removal efficiency. The repeatability of adsorption and regeneration cycles was evaluated over ten consecutive cycles for adsorbents A, B, and C, with fluctuations in efficiency maintained within ±5% relative to the initial sulphur removal capacity. This stability illustrates the stability and regeneration capacity of alumina and zeolite-based adsorbents under industrial conditions. On the contrary, adsorbent D, as expected, showed a disposable characteristic with no appreciable regeneration ability due to irreversible chemisorption of sulphur compounds. The results obtained confirm the accuracy of the measurement method and the reliability of the multi-cycle adsorption equipment used in the study.

Results

Composition and Sulphur Content of PPF before Purification

A study on sulphur removal from PPF obtained from the catalytic cracking process gave significant results, confirming the effectiveness of adsorption methods for desulphurization. Four different adsorbents (A, B, C and D) were used in the experiments, which provided a high degree of purification of PPF from various sulphur-containing compounds as well as water. Component analysis conducted before purification revealed a typical composition of PPF, optimized for high propylene yields. This characteristic enhances its value for polymerization and petrochemical synthesis processes (Table 2). Although the high propylene content confirms the commercial importance of this fraction, the presence of sulphur limits further utilization. The limitations are because sulphur and its compounds can adversely affect the catalysts in the polymerization of propylene from the fraction, so mandatory removal of sulphur-containing impurities is required to improve product quality and meet environmental standards.

Table 2: Analysis of PPF composition

Substance	*Number, %*
Ethane	1.54 wt
Ethylene	0.18 wt
Propane	16.53 wt
Propylene	81.49 wt
C_4S	0.26 wt
Sulphur (S)	5.67 ppm

Efficiency of Adsorbents in Sulphur Removal

A two-stage purification process using fixed-bed adsorbers was used to reduce sulphur levels. The first step included adsorbents A, B and C, which effectively removed impurities such as water, mercaptans and COS. The second step was to further purify the remaining traces of sulphur compounds using adsorbent D, which was designed to capture H_2S and other compounds. Table 3 illustrates the changes in PPF composition before and after purification.

Table 3: Composition analysis of purified PPF

Substance	*Before treatment, ppm*	*After treatment, ppm*
Water	290	<1
H_2S	5	<2
Mercaptans	14	<2
COS	18	<0.1

As can be seen from the table, the content of water, H_2S, mercaptans and COS was significantly reduced. Initially, the water concentration in the PPF was 290 ppm, which could adversely affect further processes because the presence of water increases the risk of corrosion and reduces the hydrocarbon separation efficiency. After adsorption treatment, the water content was reduced to less than 1 ppm, which creates the necessary

conditions for further qualitative separation of propane and propylene. However, the main challenge in this process was to remove sulphur from the PPF, as the presence degrades product quality and adversely affects the catalysts used in further processing. Adsorption methods were aimed at removing sulphur-containing compounds present in the stream in the form of hydrogen sulphide, mercaptans and COS.

Hydrogen sulphide and mercaptans, although sources of sulphur, are relatively easy components to remove (Bekbayev *et al.*, 2024; Hamidova and Samedova, 2024). In the case of H_2S, its binding to adsorbents is due to its chemical and physical properties, which allow the adsorbents to effectively capture and retain these molecules. This is how the concentration of H_2S was reduced to less than 2 ppm. Mercaptans also contain sulphur and were successfully recovered in the process due to the high selectivity of the adsorbents to these molecules. The concentration of mercaptans, which was at 14 ppm, was reduced to less than 2 ppm, which is important in terms of reducing toxicity and eliminating the pungent odour of these compounds. In addition, mercaptans can cause corrosion, making their removal necessary to improve processing conditions and equipment life. For COS, the task was more difficult due to the persistence of its structure and low reactivity. However, it was possible to significantly reduce the content of this component from 18 ppm to less than 0.1 ppm, which confirmed the high selectivity of the material. COS is one of the most resistant to the removal of sulphur-containing components, and its efficient extraction indicates the high level of adsorption technologies applied in the study.

Comparative Evaluation of Adsorbent Properties

The main contribution to purification was made by adsorbents A and C, possessing high surface area and effective adsorption centres for absorption of mercaptans and COS. In turn, adsorbent D, based on metal oxides, showed high selectivity in the removal of hydrogen sulphide, which allowed achieving the required level of purity of the product. It is important to note that adsorption materials containing metal oxides, such as D, have special active centres providing chemical interaction with H_2S, thus achieving more effective binding. The characteristics of the adsorbents used, such as granule size, bulk density, active composition and appearance, are summarized in table 4.

Table 4: Characteristics of adsorbents

Adsorbent	*Granule size, mm*	*Density, kg/m^3*	*Active ingredient*	*Appearance*
A	1.9	673	Alumina (clay-based)	White, odourless, granular
B	7x14	670	Modified alumina with zeolite	White, odourless, granular
C	7x12	785	Alumina	Grey, odourless, granular
D	7x14	801	MnO_2	Black, odourless, granulated

Alumina adsorbents (A, B and C) showed high efficiency in removing water and a number of sulphur-containing components such as mercaptans and hydrogen sulphide.

The granule size varies from 1.9 mm for adsorbent A to 7×14 mm for adsorbents B and D. Alumina, with its developed porous structure and high specific surface area, provides good adsorption through physical sorption, which is particularly effective for low molecular weight components, as in the case of H_2S and mercaptans. In addition, the high specific surface area contributes to an increased number of active centres for impurity binding, improving the overall process efficiency. Large pellets, as in B and D, have the advantage of preventing rapid pressure drop, especially when treating large volumes of gas, improving the overall stability of the adsorption process.

Adsorbent B, modified alumina with zeolite, showed improved sorption properties towards water and mercaptans due to the introduction of zeolite. Zeolites are known for their structural selectivity and ion exchange ability, which makes them particularly effective in capturing water molecules from hydrocarbon streams (Tikhonova *et al.*, 2008; Tkach *et al.*, 2021). This is due to the strong hydrophilic nature of zeolite channels, which help attract and retain water molecules, reducing water content to less than 1 ppm.

The bulk densities of adsorbents range from 670 kg/m^3 for B to 801 kg/m^3 for D. The high density, like that of adsorbent D, is due to its MnO-based composition$_2$, which has a dense structure and often provides high granule strength. Adsorbent D is designed to capture more persistent sulphur-containing compounds that are more difficult to remove by physical adsorption alone. Metal oxides have both physical and chemical adsorption mechanisms, making them particularly effective for complex molecules such as COS, which are difficult to remove due to the strong bonding of sulphur to carbon (Shevko *et al.*, 2017; 2021). In this case, adsorbent D was able to reduce the COS concentration to less than 0.1 ppm, indicating its highly selective ability to capture this particular component. In addition, the black colour and high density of the metal oxide granules indicate the ability to chemically interact, which also contributes to the efficient adsorption of more complex sulphur-containing compounds. Thus, the combination of different types of adsorbents allows effective removal of all key impurities.

Regeneration of Adsorbents Using Methane-Hydrogen Mixture

The process of regeneration of adsorbents (A, B and C) after completion of the adsorption cycle was necessary to restore their sorption capacity, because in the process of capturing sulphur and other impurities, sulphur and water molecules accumulate, which block the active surfaces of adsorbents. The use of methane as a regeneration gas was an important factor because methane, having a low molecular weight and inertness, safely interacts with adsorbents at high temperatures and effectively displaces compounds from active sites. The additional presence of hydrogen in the regeneration gas enhanced desorption of sulphur compounds, as hydrogen interacts with sulphur to form easily desorbed compounds such as sulphides.

A gradual increase in temperature to 250°C at a rate of less than 50°C per hour was necessary to prevent thermal degradation and damage to the structure of the adsorbents. Smooth heating allowed the physical and chemical bonds between adsorbed molecules and the adsorbent surface to be gently broken, preventing the abrupt release of accumulated compounds that could lead to overpressure and loss of regeneration gas. Maintaining the temperature at 230°C for 3 hours ensured complete desorption of

sulphur compounds, water and hydrocarbons, which is critical for recovery of adsorbent activity. This temperature is sufficient to break most of the bonds with sulphur compounds, but does not cause undesirable changes in the structure of alumina-based adsorbents.

After desorption, the exhaust methane gas was cooled in a heat exchanger where hydrocarbons and water were condensed. This step allowed the removal of residual impurities, recovering the regeneration gas, which was returned to the system, providing recirculation. This regeneration scheme, using a heat exchanger and closed loop, reduces regeneration gas losses, reduces emissions to the environment and lowers the overall cost of the process, making the process more sustainable and economically viable. In addition to the obvious environmental benefits of reducing the sulphur content of hydrocarbon streams, adsorption-based scrubbing has environmental trade-offs that require careful evaluation. Regeneration of adsorbents with methane at high temperatures poses the risk of methane slippage and associated greenhouse gas emissions, especially if leaks occur in a closed loop system or gas management process (Ismanzhanov, Murzakulov and Azimzhanov, 2012; Shahini *et al.*, 2023). In addition, the energy required to heat the regeneration gas to 250°C results in indirect CO_2 emissions, especially if the energy source is fossil fuel. These variables can partially negate the environmental benefits of desulfurization.

To meet overarching sustainability goals, emission reduction strategies should include improving system integrity through advanced leak detection technologies, integrating energy recovery units to utilize waste heat in the regeneration cycle, and utilizing renewable or low-carbon energy sources for heating purposes (Pasichnyk *et al.*, 2024; Vessally *et al.*, 2024). In addition, life cycle studies of non-renewable adsorbent D are needed to assess its long-term environmental impact and to explore opportunities for recycling or safe disposal. The implementation of these approaches will improve the environmental performance of the process and facilitate its scalability in the face of ever-tightening environmental regulations.

Field tests confirmed that temperature and pressure parameters have a significant effect on the efficiency of both adsorption and regeneration of adsorbents used for the removal of sulphur-containing compounds. During the experiments, it was observed that increasing the temperature to 250°C increased the desorption rate, allowing for more efficient removal of accumulated sulphur-containing molecules, speeding up the regeneration process and reducing the overall treatment time. This increase in temperature promotes a more complete breakdown of the weak chemical and physicochemical bonds between the adsorbent and the sulphur compounds, which is critical to maximize the recovery of the sorption capacity of the material. The temperature of the adsorbent regeneration process cannot be increased above 250°C for several reasons related to technical, economic and operational constraints. At higher temperatures, many adsorbents, such as alumina and zeolite materials, begin to lose their structural strength, which can lead to deformation of the porous structure and a decrease in sorption capacity (Adekenov and Gafurov, 1992; Alkaabi, Obaid and Abdulsada, 2022). In addition, higher temperatures can contribute to changes in the phase composition of the active components, reducing the selectivity of adsorbents to sulphur-containing compounds. In addition, high temperatures increase the risk of uncontrolled

desorption of hydrocarbons, which causes saturation of the regeneration gas with hydrocarbon impurities and complicates their subsequent condensation and utilization. The increased fire hazard must also be taken into account: high temperatures increase the probability of ignition of the gas mixture, especially in the presence of methane and hydrogen, which requires strict control.

The system pressure also had a significant effect on the process efficiency. At the operating pressure of 0.5 MPa, the adsorbents showed maximum efficiency in sulphur removal. Higher pressure, providing a tighter contact between the gas phase and the adsorbent, increases the number of molecules of sulphur-containing compounds that can contact the active surface of the adsorbent per unit time (Lyubchik *et al.,* 2005; Turmukhambetov *et al.*, 1999). This factor, especially at high partial pressure of sulphur compounds, facilitates their capture by the adsorbent and subsequent desorption at elevated temperature. The study confirmed the high efficiency of adsorption methods for the removal of sulphur-containing compounds from PPF. The use of four different adsorbents in a two-stage purification process significantly reduced the content of water, mercaptans, COS and hydrogen sulphide to acceptable levels, making the PPF suitable for further processing. The adsorbent regeneration process using methane demonstrated its economic feasibility and sustainability. The approach provided regeneration and reuse of adsorbents up to 8-10 cycles with minimal loss of efficiency.

Scaling up the adsorption-based desulfurization process to full industrial capacity involves numerous practical obstacles that require careful control. A significant problem is the need for frequent replacement of non-regenerable adsorbent D (MnO_2), which, despite its high selectivity for residual COS and H_2S, requires periodic recycling and replenishment, resulting in increased operating and environmental costs. The use of methane for high-temperature regeneration of adsorbents A, B and C is successful but requires significant energy inputs, mainly due to the need for prolonged heating to 250°C and associated cooling and gas recirculation systems. This raises concerns about energy efficiency and environmental pollution, especially at large sizes. In addition, the use of large pellets, especially in B and D adsorbents, although favourable for reducing bed pressure drop and improving flow stability, can hinder adsorption kinetics due to reduced surface area, requiring longer contact times or increased reactor volume. To address these issues, scale-up plans should include optimization of the adsorbent form, evaluation of alternative energy recovery systems, and development of cost-effective procedures for recycling or reactivation of MnO_2-based materials.

Discussion

The results of the study show that adsorption is an effective method for the removal of sulphur-containing compounds from PPF obtained in the catalytic cracking process. The use of four different adsorbents (A, B, C and D) achieved a significant reduction in the concentrations of water and sulphur compounds. The obtained data confirm the results of previous studies, in which adsorption methods demonstrated high efficiency in the removal of sulphur-containing impurities from hydrocarbon streams. In this study, adsorbent B containing zeolite showed high efficiency in reducing the concentration of hydrogen sulphide and mercaptans to less than 2 ppm. The results confirm previous studies such as the work of Lozinska-Miller *et al.* (2020), in which Na-Y and La-Y

zeolites showed high selectivity to sulphur-containing compounds, making them suitable for desulphurization. However, in contrast to the proposed approach, modified zeolites, as shown in the study by Othman, Salih and Hamasalih (2022), showed even higher efficiency, especially for the removal of complex sulphur-containing molecules such as 4,6-dimethyldibenzothiophene and dibenzothiophene. According to this, modification of zeolites can significantly improve their adsorption properties. For example, in the work of Jangi, Rahemi and Allahyari (2022), the use of phosphorus-tungstic acid on different natural zeolites also showed significant removal of dibenzothiophene, where phosphorus-tungstic acid-Ferr (phosphorus-tungstic acid – ferrierite) achieved removal up to 82.02% under optimized conditions. This study also obtained impressive results; however, the specificity of the adsorbents used and the sulphur removal process differed. In addition, Zhang *et al.* (2023) showed that modified NaY zeolites containing alcohol groups provided dynamic adsorption of sulphur compounds at 315 mg/g.

The obtained data are also in agreement with the results of Ghassa *et al.* (2023), where activated carbon was used in the adsorption of sulphur-containing compounds. In published studies, activated carbon showed high adsorption capacity for sulphur impurities such as H_2S and COS due to its large specific surface area and developed porous structure. In this study, alumina-based adsorbent C showed comparable efficiency to activated carbon, indicating that alumina-based adsorbents can be an effective alternative to activated carbon in removing sulphur-containing compounds from hydrocarbon streams. It is interesting to note that the MnO-based adsorbent D used performed better than activated carbon in terms of COS removal, reducing its concentration to less than 0.1 ppm. Metal oxides such as ZnO are widely used in adsorption systems for sulphur removal. Dogan *et al.* (2022) note that their high efficiency is due to the chemical reaction of sulphur-containing compounds with the active surfaces of oxides. This indicates the promising use of such adsorbents for highly selective removal of COS, which is also confirmed by the results of this study. Such efficiency is also confirmed by other studies. For example, in the study of Lyu *et al.* (2023), an initial sulphur removal efficiency of 94.5% was achieved on a fresh oxide adsorbent, and the sulphur content at the outlet dropped below 10 μg/g.

The adsorbents used in this study showed different efficiency depending on their composition and structure. Adsorbent A, containing alumina, showed high efficiency in removing water from the PPF, which is consistent with the known properties of alumina, often used for the dehydration of gases and hydrocarbon streams. These results support the feasibility of using alumina for moisture removal in PPF purification. In another study by Mahesh *et al.* (2023), the authors noted the key features of Al_2O_3 and its composites that make it ideal for impurity removal: high specific surface area, porous structure and the presence of numerous active centres. Moreover, aluminium oxide is often used in nanomaterials for water purification due to its metastable phases and hexagonal structure, which significantly enhance its adsorption properties. Thus, the observed efficiency of adsorbent A in this study is consistent with these characteristics of aluminium oxide, making it a versatile and effective solution for the adsorptive treatment of both water and other pollutants.

Adsorbent B, a modified zeolite, demonstrated high efficiency in the removal of hydrogen sulphide and mercaptans. This confirms the results of other studies, where

zeolites show high selectivity to organic sulphur-containing compounds due to their porous structure and active centres. As in the study of Li *et al.* (2024b) on mercaptan adsorption, the results showed that micropore filling and chemical binding are the main adsorption mechanisms. Thus, mesoporous zeolite showed that its modification significantly increased the surface area to 73.7 m^2/g and the total pore volume to 0.107 cm^3/g. The impregnated samples showed better adsorption characteristics compared to the original zeolite, noted Maghsoudi *et al.* (2023). The advantage of zeolites, as stated by Zewdie, Bizualem and Nurie (2024), is their ability to effectively remove sulphur-containing compounds even at relatively low concentrations, which makes them attractive for use as adsorbents in desulphurization processes.

Adsorbent C, based on pure alumina, showed high performance in removing both moisture and sulphur compounds. This confirms the versatility of alumina as an adsorbent capable of handling different types of pollutants. However, its efficiency compared to zeolites was slightly lower due to its lower selectivity for sulphur-containing compounds. Adsorbent D, MnO_2, showed the highest efficiency for the removal of COS and H_2S. Metal oxides such as zinc oxide are known for their ability to bind to sulphur compounds by chemical sorption. The result is in line with previous findings described in the work by Georgiadis, Charisiou and Goula (2020) and Botin *et al.* (2023), where metal oxides were used to effectively remove sulphur from various hydrocarbon streams.

The adsorption efficiency was also affected by the process conditions, such as temperature and pressure. In this study, PPF was fed to the adsorbers at a temperature of 15°C and a pressure of 2.8 MPa. These parameters were found to be optimal for the removal of sulphur-containing compounds and moisture. The results showed that even at relatively low temperatures, the adsorbents showed high efficiency due to the favourable conditions for physical and chemical adsorption. In addition, in a study by Zheng *et al.* (2024), increasing temperature and pressure favoured the removal of sulphur compounds, while the optimum conditions of this experiment also included low temperatures for maximum efficiency. Adsorption desulphurization technology, as described in the review of Li *et al.* (2024a), has significant advantages compared to the traditional Merox method: it is more cost-effective, simpler and environmentally friendly, besides, it can be carried out even at room temperature (20-25°C). The Merox method uses a catalyst based on transition metal salts that promote the oxidation of mercaptans to disulphides, which facilitates their removal from the hydrocarbon feedstock. However, the process is carried out at higher temperatures (30-70°C) to reduce energy costs and environmental damage. The study conducted by Li *et al.* also emphasizes the high efficiency of zeolite-based adsorbents for the removal of mercaptans from petroleum fractions, which is in line with the expectation of method substitution. In contrast to the adsorbents mentioned in the review by Li *et al*., the study demonstrated a significant reduction in the concentration of sulphur-containing compounds even at relatively low temperatures.

In the review by Renu and Sithole (2024), the authors examined various methods of adsorbent regeneration, including chemical, thermal, microwave, electrochemical, and ultrasonic techniques, achieving maximum desorption efficiencies of up to 99.5% for chemical regeneration. Shah, Pre, and Alappat (2013) note that while conventional

methods involve high operating costs and waste generation, this study utilised a mixture of methane and hydrogen as regeneration gas, enhancing the efficiency and sustainability of the adsorbent regeneration process following the removal of sulphur-containing compounds. This approach demonstrated the economic viability of using these adsorbents, as they can be reused multiple times without a significant loss of efficiency. Another critical factor is the maintenance of temperature in adsorbers during regeneration. In this context, regeneration at 230ºC proved highly effective. This finding aligns with previous studies by Kulkarni and Kaware (2014), which indicated that regenerating adsorbents such as zeolites and activated carbon necessitates controlled heating to restore their adsorption capacity without compromising the adsorbent structure.

This study confirms the high efficiency of adsorption methods for the removal of sulphur-containing compounds from PPFs. Comparison with previous studies, such as those of Pérez-Botella, Valencia, and Rey (2022) and Haruna *et al.* (2022), showed that the results are comparable to those already known for the use of zeolites, alumina, and metal oxides for hydrocarbon desulphurization. Adsorbent D containing MnO_2, which showed high selectivity to COS and H_2S, was found to be particularly promising. Successful regeneration of adsorbents using methane confirms the economic feasibility of the proposed process.

Conclusions

In this study, four different adsorbents (A, B, C and D) were investigated to test the effectiveness of adsorption methods for the removal of sulphur-containing impurities in the PPF obtained from the catalytic cracking process. Field tests have shown at the Ethylene-Polyethylene plant (Sumgait, Azerbaijan) that application of a two-stage adsorption purification process allows successful removal of such sulphur-containing compounds as water, mercaptan, COS, AsH_3 and H_2S from the C3-hydrocarbon stream. The initial sulphur content of the PPF feedstock was 5.67 ppm, but after treatment, it was reduced to less than 2 ppm, which confirms the high efficiency of the proposed purification process and the use of these adsorbents. Reduction of sulphur content to such values contributes to the protection of catalysts and equipment used in the further propylene polymerization process, which avoids efficiency losses and increases equipment lifetime.

This study allowed the evaluation of the basic properties of adsorbents and their applicability for PPF desulphurization, and demonstrated their regeneration ability. The use of methane for adsorbent regeneration showed good results, indicating the stability of the adsorbents and the economic viability of the proposed method. Further studies are recommended to optimize the desulphurization conditions, such as pressure, temperature and adsorbent composition, to improve the process efficiency. Additional study of the influence of operating parameters on adsorbent performance may also contribute to process improvement, including energy efficiency and cost reduction. The results of this study highlight the potential of adsorption as a promising method for sulphur removal from hydrocarbon streams, which not only improves product quality, but also reduces environmental impact and raises environmental standards in the petrochemical industry. Thus, this adsorptive desulphurization method using a two-stage approach represents an

important step towards more sustainable and efficient hydrocarbon feedstock treatment technologies that meet modern industrial and legislative requirements.

The results show that increasing the regeneration temperature to 250°C markedly increases the desorption efficiency of sulphur-containing compounds from regenerated adsorbents A, B and C, while maintaining their structural integrity. Maintaining an adsorption pressure of 2.8 MPa during treatment enhances mass transfer of contaminants to the adsorbent surface, while a regeneration pressure of 0.5 MPa promotes efficient gas desorption. A hypothetical slight increase in adsorption temperature to 25-30°C could improve the capture kinetics of sulphur components without compromising hydrocarbon integrity. Future optimization studies recommend investigating the interaction between residence time, adsorbent bed delamination, and intermediate pressure, which could lead to improved impurity capture and longer adsorption cycle times. It is recommended that pressure swing adsorption (PSA) or temperature-programmed regeneration (TPR) methods be investigated to increase adsorbent lifetime and minimize downtime.

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Authors' Declarations and Essential Ethical Compliances

Authors' Contributions (in accordance with ICMJE criteria for authorship)

Contribution	*Author 1*	*Author 2*
Conceived and designed the research or analysis	Yes	No
Collected the data	No	Yes
Contributed to data analysis & interpretation	Yes	Yes
Wrote the article/paper	Yes	Yes
Critical revision of the article/paper	Yes	No
Editing of the article/paper	No	Yes
Supervision	Yes	No
Project Administration	Yes	No
Funding Acquisition	No	No
Overall Contribution Proportion (%)	50	50

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Research involving human bodies or organs or tissues (Helsinki Declaration)
The author(s) solemnly declare(s) that this research has not involved any human subject (body or organs) for experimentation. It was not a clinical research. The contexts of human population/participation were only indirectly covered through literature review. Therefore, an Ethical Clearance (from a Committee or Authority) or ethical obligation of Helsinki Declaration does not apply in cases of this study or written work.

Research involving animals (ARRIVE Checklist)
The author(s) solemnly declare(s) that this research has not involved any animal subject (body or organs) for experimentation. The research was not based on laboratory experiment involving any kind animal. The contexts of animals were only indirectly covered through literature review. Therefore, an Ethical Clearance (from a Committee or Authority) or ethical obligation of ARRIVE does not apply in cases of this study or written work.

Research on Indigenous Peoples and/or Traditional Knowledge
The author(s) solemnly declare(s) that this research has not involved Indigenous Peoples as participants or respondents. The contexts of Indigenous Peoples or Indigenous Knowledge were only indirectly covered through literature review. Therefore, an Ethical Clearance (from a Committee or Authority) or prior informed consent (PIC) of the respondents or Self-Declaration in this regard does not apply in cases of this study or written work.

Research involving Plants
The author(s) solemnly declare(s) that this research has not involved the plants for experiment and field studies. Some contexts of plants are also indirectly covered through literature review. Thus, during this research the author(s) obeyed the principles of the Convention on Biological Diversity and the Convention on the Trade in Endangered Species of Wild Fauna and Flora.

Research Involving Local Community Participants (Non-Indigenous) or Children
The author(s) solemnly declare(s) that this research has not directly involved any local community participants or respondents belonging to non-Indigenous peoples. Neither this study involved any child in any form directly. The contexts of different humans, people, populations, men/women/children and ethnic people were only indirectly covered through literature review. Therefore, an Ethical Clearance (from a Committee or Authority) or prior informed consent (PIC) of the respondents or Self-Declaration in this regard does not apply in cases of this study or written work.

PRISMA (Preferred Reporting Items for Systematic Reviews and Meta-Analyses)
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